

What is claimed is:

1                   1.     A method of making a hexammine cobaltic salt, the method  
2     comprising:

3                   (a) providing a reaction vessel containing an aqueous solution of at least  
4     one ammonia source selected from the group consisting of ammonium hydroxide,  
5     ammonia-containing gas and combinations thereof;

6                   (b) introducing a solution of  $\text{Co}(\text{X})_2$  and a solution of  $\text{NH}_4\text{X}$ , wherein  
7     X is at least one selected from the group consisting of chloride, bromide, perchlorate,  
8     and nitrate, into the reaction vessel to form reaction vessel contents comprising a first  
9     product precursor;

10                  (c) introducing an oxygen-containing gaseous oxidizer to the reaction  
11     vessel contents of (b) to form an at least partially oxidized second product precursor;

12                  (d) heating the at least partially oxidized second product precursor to  
13     form a third product precursor;

14                  (e) adding a surface active catalyst to the third product precursor;

15                  (f) continue introducing an oxygen-containing gaseous oxidizer to the  
16     catalyst-containing reaction mixture of (e) while maintaining the temperature at a  
17     temperature selected to result in product formation of a desired particle size; and

18                  (g) cooling the mixture from (f) and recovering the hexammine cobaltic  
19     salt therefrom as an admixture with the surface active catalyst.

2. The method of claim 1 wherein X is nitrate and the hexammine cobaltic salt is hexammine cobaltic nitrate.

3. The method of claim 2 wherein the first product precursor comprises  $\text{Co}(\text{NH}_3)_5(\text{H}_2\text{O})(\text{NO}_3)_2$ .

4. The method of claim 2 wherein the at least partially oxidized second product precursor comprises  $\mu$ -peroxobis [pentamminecobalt].

5. The method of claim 2 wherein the third product precursor comprises pentammine-aqua cobaltic nitrate.

6. The method of claim 2 wherein the at least partially oxidized second product precursor is heated in (d) to a temperature in a range of about 95°F to about 120°F.

7. The method of claim 2 wherein in (f) the temperature is maintained in a range of about 95°F to about 120°F.

1                   8.     The method of claim 1 wherein the providing of the aqueous  
2     solution of an ammonia source in the reaction vessel in (a) comprises adding the  
3     ammonia source to the reaction vessel in an amount sufficient to provide about 5.5 to  
4     about 6.5 equivalents of ammonia to cobalt.

1                   9.     The method of claim 1 wherein the solution of  $\text{Co}(\text{X})_2$  and  $\text{NH}_4\text{X}$   
2     comprises water,  $\text{Co}(\text{NO}_3)_2$  and ammonium nitrate.

1                   10.    The method of claim 1 wherein the surface active catalyst  
2     comprises activated carbon.

1                   11.    The method of claim 10 wherein the activated carbon surface  
2     active catalyst is added in an amount sufficient to provide no more than about 1.5 %  
3     carbon in the hexammine cobaltic salt.

1                   12.    The method of claim 11 wherein the activated carbon surface  
2     active catalyst is added in an amount sufficient to provide no more than about 0.5 %  
3     carbon in the hexammine cobaltic salt.

1                   13.    The method of claim 12 wherein the activated carbon surface  
2   active catalyst is added in an amount sufficient to provide about 0.2-0.3 % carbon in  
3   the hexammine cobaltic salt.

1                   14.    The method of claim 1 wherein the oxygen-containing gaseous  
2   oxidizer is introduced to the heated combination in (c) to form an at least partially  
3   oxidized reaction mixture having an absorbance @ 505 nm of at least about 1.5 AU.

1                   15.    The method of claim 1 wherein the hexammine cobaltic salt  
2   recovered in (f) is of a particle size in the range of about 35 microns to about 60  
3   microns.

1                   16.    The method of claim 1 wherein the reaction vessel contents of (b)  
2   are heated prior to the initiation of the introduction of the oxygen-containing gaseous  
3   oxidizer.

1                   17.    The method of claim 1 wherein the introduction of the solution  
2   of  $\text{Co(X)}_2$  and the solution of  $\text{NH}_4\text{X}$  comprises introducing a solution containing both  
3    $\text{Co(X)}_2$  and  $\text{NH}_4\text{X}$ .

1                   18.    A method of making a hexammine cobaltic nitrate having a  
2   particle size in the range of about 35 microns to about 60 microns, the method  
3   comprising:

4                   (a) providing a reaction vessel containing an aqueous solution of at least  
5   one ammonia source selected from the group consisting of ammonium hydroxide, an  
6   ammonia-containing gas and combinations thereof;

7                   (b) introducing a solution of  $\text{Co}(\text{NO}_3)_2$  and  $\text{NH}_4\text{NO}_3$  into the reaction  
8   vessel to form reaction vessel contents comprising a first product precursor;

9                   (c) heating the first product precursor to a temperature in the range of  
10   95°F to 120°F;

11                  (d) introducing an oxygen-containing gaseous oxidizer to the heated first  
12   product precursor in (c) to form an at least partially oxidized reaction mixture having  
13   an absorbance @ 505 nm of at least about 1.5 AU;

14                  (e) adding a surface active catalyst of activated carbon to the reaction  
15   mixture of (d);

16                  (f) continue introducing an oxygen-containing gaseous oxidizer to the  
17   catalyst-containing reaction mixture of (e) while maintaining the temperature within  
18   a range of about 95°F to about 120°F and which temperature is selected to result in  
19   to result in the hexammine cobaltic nitrate having a desired particle size; and

20                  (g) cooling the mixture from (f) and recovering the hexammine cobaltic  
21   nitrate therefrom as an admixture with the surface active catalyst.

1                    19.    The method of claim 18 wherein the providing of the aqueous  
2                    solution of an ammonia source in the reaction vessel in (a) comprises adding the  
3                    ammonia source to the reaction vessel in an amount sufficient to provide about 5.5 to  
4                    about 6.5 equivalents of ammonia to cobalt.

1                    20.    The method of claim 18 wherein the activated carbon surface  
2                    active catalyst is added in an amount sufficient to provide no more than about 1.5 %  
3                    carbon in the hexammine cobaltic salt.

1                    21.    The method of claim 18 wherein in (d), the oxygen-containing  
2                    gaseous oxidizer is introduced to the heated first product precursor in (c) to form an  
3                    at least partially oxidized reaction mixture having an absorbance @ 505 nm of about  
4                    2.5 AU.

1                   22.    A method of making a hexammine cobaltic nitrate having a  
2   selected particle size, the method comprising:

3                   (a) providing a reaction vessel containing an aqueous solution of at least  
4   one ammonia source selected from the group consisting of ammonium hydroxide,  
5   ammonia-containing gas and combinations thereof;

6                   (b) introducing a solution of  $\text{Co}(\text{NO}_3)_2$  and  $\text{NH}_4\text{NO}_3$  into the reaction  
7   vessel to form reaction vessel contents comprising  $\text{Co}(\text{NH}_3)_5(\text{H}_2\text{O})(\text{NO}_3)_2$ ;

8                   (c) introducing an oxygen-containing gaseous oxidizer to the reaction  
9   vessel contents of (b) to form  $\mu$ -peroxobis [pentamminecobalt];

10                  (d) heating the  $\mu$ -peroxobis [pentamminecobalt] to form  
11   pentammine-aqua cobaltic nitrate;

12                  (e) adding a surface active catalyst of activated carbon to the  
13   pentammine-aqua cobaltic nitrate;

14                  (f) continue introducing an oxygen-containing gaseous oxidizer to the  
15   catalyst-containing reaction mixture of (e) while maintaining the temperature at a  
16   selected temperature in the range of about 95°F to about 120°F to result in the  
17   hexammine cobaltic nitrate having a selected particle size in the range of about 35  
18   microns to about 60 microns; and

19                  (g) cooling the mixture from (f) and recovering the hexammine cobaltic  
20   nitrate therefrom as an admixture with the surface active catalyst.

1                   23.    The method of claim 22 wherein the providing of the aqueous  
2    solution of the at least one ammonia source in the reaction vessel in (a) comprises  
3    adding the ammonia source to the reaction vessel in an amount sufficient to provide  
4    about 5.5 to about 6.5 equivalents of ammonia to cobalt.

1                   24.    The method of claim 22 wherein the activated carbon surface  
2    active catalyst is added in an amount sufficient to provide no more than about 1.5 %  
3    carbon in the hexammine cobaltic salt.

1                   25.    The method of claim 22 wherein the oxygen-containing gaseous  
2    oxidizer is introduced to the heated combination in (c) to form an at least partially  
3    oxidized reaction mixture having an absorbance @ 505 nm of at least about 1.5 AU.

1                   26.    The method of claim 22 wherein the reaction vessel contents of  
2    (b) are heated prior to the initiation of the introduction of the oxygen-containing  
3    gaseous oxidizer.